UNIVERSITI PUTRA MALAYSIA

SYNTHESIS AND CHARACTERIZATION OF PALM OIL-BASED RESIN FOR UV COATING

CHEONG MEI YEE.

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By

CHEONG MEI YEE

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirement for the Degree of Master of Science

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To my family and friends,

For their unremitting support and encouragement

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

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May 2006

Chairman: Professor Wan Md. Zin B. Wan Yunus, PhD

Faculty: Science

The production of UV curable acrylated polyol esters from palm oil and its downstream products offers potential and promising materials for a number of applications such as polymeric film preparation and wood coatings. In this study, palm olein-based polyol of ethylene glycol was reacted with acrylic acid in the presence of a catalyst and inhibitors via condensation esterification process at various temperatures (70.0, 80.0, 90.0 or 100.0°C), stirring speed of 100 or 400 rpm and under vacuum for 15 hours. The reaction temperature of 80.0°C and the stirring rate of 400 rpm produce a homogeneous product. Optimizations for the synthesis were also carried out using a catalyst concentrations ranging from 0.0 to 5.0% of polyol weight.

Fifteen different formulations have been investigated using the synthesized prepolymers with monomers and a small amount of photoinitiator. Monomers used were 1, 6-hexanediol diarylate (HDDA) and trimethylolpropane triacrylate (TMPTA) while photoinitiator used was 1-hydroxy cyclohexyl phenylketone (Irgacure 184). The mixtures were cured to make thin polymeric films under UV radiation with doses between 2 to 14 passes (energy per pass is 0.600 J/cm^2). Coating and curing was carried out on glass for pendulum hardness characterization, haziness and scratch measurement as well as FT-IR analysis. Coating and curing were done on rubber wood for cross hatch adhesion test.

Pendulum hardness of the film prepared from the UV curable formulation with monomer HDDA and the prepolymer prepared using 3% catalyst was 24.5%. The radiation dose was 14 passes. The highest pendulum hardness of 49.4% was achieved when using UV curable formulation with monomer TMPTA and the prepolymer prepared using 2% catalyst. The radiation dose needed was 10 passes. Formulation of UV curable coating using the optimized acrylated polyol ester prepolymer and monomer TMPTA exhibited higher scratch measurement and lower haziness compared to those of the formulation with monomer HDDA. UV curable formulation of prepolymer with monomer HDDA gave better adhesion on rubber wood than that of with monomer TMPTA. Films cured by UV radiation made from prepolymers on both glass panel and rubber wood surfaces showed good hardness, scratch resistance and adhesion with almost clear transparency. In general, it can be concluded that newly synthesized UV radiation curable palm-based acrylated polyol ester prepolymers are promising candidates for wood coating applications.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

SINTESIS AND PENCIRIAN RESIN BERASASKAN MINYAK SAWIT UNTUK PENGLITUPAN MATANG UV

Oleh

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Mei 2006

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Prapolimer poliester akrilat boleh matang UV yang disediakan daripada sumber minyak sawit dan terbitan hasilnya mempunyai potensi dan harapan untuk kegunaan bahan bukan makanan seperti pembuatan filem polimerik untuk litupan kayu. Dalam kajian ini, produk berasaskan minyak sawit iaitu poliol ditindakbalas dengan asid akrilik melalui proses esterifikasi dengan kehadiran mangkin dan perencat gel pada suhu berlainan (70.0, 80.0, 90.0 atau 100.0°C), kadar adukan; 100 rpm atau 400 rpm dan dalam keadaan vakum selama 15 jam. Suhu tindak balas 80.0°C dan kadar adukan 400 rpm menghasilkan produk homogen. Optimasi penyediaan dilakukan dengan menggunakan mangkin dalam linkungan 0.0 hingga 5.0% daripada berat poliol.

Kajian dilakukan untuk lima belas formulasi prapolimer yang disintesis bersama monomer dan sedikit bahan fotopemula. Monomer digunakan ialah 1, 6-heksanadiol diakrilat (HDDA) dan trimetilolpropana triakrilat (TMPTA) manakala bahan fotopemula ialah 1-hidroksi sikloheksil fenilketon (Irgacure 184). Campuran dimatangkan di bawah sinaran ultralembayung (UV) pada pelbagai dos di antara 2 hingga 14 laluan di mana tenaga per laluan ialah 0.600 J/cm^2 untuk menghasilkan filem polimer yang nipis. Litupan dan pematangan oleh UV dilakukan pada permukaan kaca untuk pencirian kekerasan, ukuran kelutsinaran, ujian tahan calar dan analisis FT-IR. Litupan dan pematangan UV juga dilakukan pada kayu untuk ujian daya kelekatan.

Kekerasan filem yang disediakan daripada formulasi matang UV dengan monomer HDDA dan prapolimer yang disediakan dengan menggunakan 3% mangkin ialah 24.5%. Dos radiasi yang diperlukan ialah 14 laluan. Kekerasan yang paling tinggi ialah 49.4% terhasil dengan menggunakan formulasi matang UV dengan monomer TMPTA dan prapolimer yang disediakan menggunakan 2% mangkin. Sinaran dos yang diperlukan ialah 10 laluan. Formulasi matang UV yang terdiri daripada prapolimer teroptima dan monomer TMPTA memberi ukuran tahan calar dan kelutsinaran yang tinggi manakala formulasi prapolimer dan monomer HDDA memberi daya kelekatan yang lebih tinggi pada kayu getah. Pada keseluruhannya, filem yang diperbuat oleh prapolimer yang telah dimatangkan oleh UV menunjukkan kekerasan, tahan calar, daya kelekatan dan kelutsinaran yang baik. Kesimpulannya, prapolimer poliester akrilat boleh matang UV yang disediakan daripada sumber minyak sawit berpotensi digunakan untuk litupan kayu.

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I certify that an Examination Committee has met on 17th of May 2006 to conduct the final examination of Cheong Mei Yee on her Master of Science thesis entitled "Synthesis and Characterization of Palm Oil-Based Resin for UV Coating" in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The Committee recommends that the candidate be awarded the relevant degree. Members of the Examination Committee are as follows:

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This thesis submitted to the Senate of Universiti Putra Malaysia has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of the Supervisory Committee are as follows:

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DECLARATION

I hereby declare that the thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at UPM or other institutions.

CHEONG MEI YEE

Date: 11/9/2006

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES